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Standard Specification for Minimum Requirements for Laboratories Engaged in Chemical Analysis of Soil, Rock, and Contained Fluid[1]

This standard is issued under the fixed designation D 5522; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope *

1.1 This specification covers specific criteria for evaluating the technical capabilities of laboratories involved in testing, measuring, inspecting, and calibrating activities related to chemical analysis of earth materials. In this specification, earth materials shall mean soil, rock, and contained fluids. For the sake of brevity, the term "laboratory" is used in this practice to represent all the above.

1.2 This specification addresses the minimum requirements for laboratories that analyze earth materials for metals, volatile organic compounds, semivolatile organic compounds, pesticides, herbicides, PCBs, radionuclides, and various other parameters by miscellaneous wet chemistry techniques.

1.3 This specification presents specific criteria to be used in an evaluation, including restrictions, minimum requirements, and benchmarks of compliance for specific tests or for specific types of tests.

1.4 This specification is meant only for the evaluation of facilities performing chemical analysis of earth materials and is in no way intended to be an absolute guide. It shall not replace specific criteria that exist for test methods or that exist as separate standards. In instances where laboratory evaluation sections are included as part of a test method, or where specific criteria for test methods exist as separate standards, those separate criteria should also be considered.

1.5 Minimum requirements for agencies engaged in the physical testing of soil and rock can be found in Practice D 3740.

1.6 The values stated in SI units are to be regarded as the standard.

1.7 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 3740 Practice for Minimum Requirements for Agencies Engaged In the Testing and/or Inspection of Soil and Rock as Used In Engineering Design and Construction[2]

2.2 *USEPA Publications:*

SW-846 Test Methods for Evaluating Solid Waste[3]

Methods for the Examination of Water and Wastewater[2]

3. Summary of Specification

3.1 This specification covers minimum requirements for the following items:

3.1.1 Organization of the laboratory and its affiliates,
3.1.2 Personnel,
3.1.3 Quality system,
3.1.4 Facilities and equipment,
3.1.5 Calibration,
3.1.6 Test methods and procedures,
3.1.7 Records,
3.1.8 Test reports, and
3.1.9 Standard operating procedures.

3.2 The items listed here as criteria to be reviewed during an evaluation are standard items that the laboratory shall be following. This includes items that shall be available during an assessment and that the laboratory personnel shall be able to show are being completed for each analysis type.

4. Significance and Use

4.1 This specification is meant for use when evaluating laboratories engaged in chemical analysis of earth materials.

4.2 The criteria specified in this specification can be used in the process of accreditation.

5. Organization

5.1 The legal name, address, and telephone number of the laboratory must be available.

5.2 An organization chart that shows the following information must be presented in the quality control manual:

5.2.1 Ownership or membership,
5.2.2 Names of affiliations,
5.2.3 Management structure,
5.2.4 Principal officers,
5.2.5 Directors,
5.2.6 Relevant organizational components, and

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[2] *Annual Book of ASTM Standards,* Vol 04.08.

[3] Available from United States Environmental Protection Agency.

***A Summary of Changes section appears at the end of this standard.**

5.2.7 Principal chemists.

5.3 Conflicts of interest among the various affiliations must be noted in the evaluation. Such conflicts of interest should be avoided by the laboratory.

5.4 External and subcontracted technical services must be named by the laboratory, along with the addresses and contacts. The subcontractors must have undergone an assessment by the laboratory and documentation of the findings must be available. These outside technical services must meet the requirements outlined in this specification for the relevant test procedures or for specific types of tests performed by the subcontractor for the primary laboratory.

5.5 The quality-control manual must contain a description of its facilities and a summary of the scope of operations.

5.6 Key management and supervisory personnel in each relevant operating, support, and service unit in the laboratory's functional organization and the reporting relationships must be identified and a job description for each of these personnel included as part of the quality-control manual. Resumes on each of these individuals must be included. Each of these individuals shall understand the extent of their responsibilities.

5.7 Qualifications, accreditations, and recognition of the laboratory by others shall be presented, along with a copy of the certifications, in the quality-control manual.

6. Personnel

6.1 The quality-control manual shall address the means by which all personnel will be trained. The manual shall also address the means by which the records will be maintained for said training and work experience.

6.2 All personnel must undergo an introduction to the quality-control manual as well as to the test procedures for which they will be responsible. Appropriate documentation of this training shall be available for review. This documentation shall include any reviews along with the date of the review, a listing of in-house training and certifications from outside training courses, and documented evidence of the analyst's proficiency for each test method performed. This documentation shall be available for review upon request. No analyst shall perform a given test procedure without the appropriate training.

6.3 Each person with the following duties or titles must meet or be supervised by a person who meets the specified minimum experience or have an appropriate educational background:

6.3.1 Atomic absorption/ICP supervisor, two years,

6.3.2 Atomic absorption analysis, one year,

6.3.3 Atomic absorption/ICP sample preparation, three months,

6.3.4 Gas chromatography supervisor, two years,

6.3.5 Gas chromatography analysis, six months,

6.3.6 Gas chromatography spectral interpretation, two years,

6.3.7 Purge and trap analysis (GC), six months,

6.3.8 Extraction and concentration expert, one year,

6.3.9 PCB and pesticide residue analysis expert, two years,

6.3.10 General chemistry and instrumentation, six months,

6.3.11 GC/MS supervisor, two years,

6.3.12 GC/MS operator, four months full-time,

6.3.13 GC/MS spectral interpretation, two years,

6.3.14 Purge and trap analyst (GC/MS), six months,

6.3.15 Microbiology supervisor, one year,

6.3.16 Radiochemistry supervisor, five years,

6.3.17 Radionuclides analyst, one year,

6.3.18 Gross alpha/beta technician, six months,

6.3.19 Visible spectroscopy supervisor, two years,

6.3.20 Visible spectroscopist, one year,

6.3.21 Spectral interpretation (visible spectroscopy), two years, and

6.3.22 Inorganic sample preparation—3 months.

7. Quality System

7.1 The quality system must be documented in a manual or equivalent.

7.2 The quality manual must be available to all personnel.

7.3 The quality manual shall be updated at least annually, but will always be under revision.

7.4 The quality manual shall contain the following:

7.4.1 Organizational charts,

7.4.2 Staff duties including responsibilities for quality,

7.4.3 Feedback and corrective action program for internal problems,

7.4.4 Technical complaint handling procedure,

7.4.5 Policy for documenting procedures and analysis methods,

7.4.6 Procedure for sample collection and preservation if performed by laboratory personnel,

7.4.7 Procedure for sample storage and handling,

7.4.8 Quality-control requirements for each type of test,

7.4.9 Procurement and inventory procedures,

7.4.10 Policy on the operation and calibration of instruments,

7.4.11 Policy on preventative maintenance,

7.4.12 Procedure for record keeping and record storage,

7.4.13 Procedure for checking the reliability of data reduction and reporting,

7.4.14 Procedure for correcting erroneous reports, and

7.4.15 Record retention policy.

7.5 The quality-assurance (QA) manager shall have direct access to top management and operate independently of the rest of the laboratory.

7.6 The QA manager should have the power to oversee the laboratory procedures, identify problems, and make recommendations to management.

7.7 A method shall exist so that any deviations or deficiencies in QC are reported to management and such reports are documented.

7.8 All new employees must be given a copy of the quality manual and be required to read it.

7.9 All employees must be given a copy of any changes or additions to the quality manual and be required to review the manual at least once per year.

7.10 All personnel must be trained or prove proficiency for any test method they perform. The training can either be provided in-house or by a certified training school.

7.11 At least quarterly, the QA manager or designee shall conduct an internal audit of the laboratory and report his findings to the laboratory director.

7.11.1 Any problems discovered during the quarterly audit shall be corrected and the steps taken to correct the problem documented.

7.12 All supervisory staff should be aware of the QA/QC system and its application to the daily activities of the laboratory.

7.13 Standard curves that adequately cover the expected sample concentration ranges shall be prepared at least annually or more often as required by the specific test methods. A new curve shall also be prepared when new reagents are prepared.

7.14 Standard curves shall be prepared with a minimum of three standards and one blank or as specified by the method.

7.15 A procedure shall exist such that records indicate what corrective action has taken place when analytical results fail to meet QC criteria.

7.16 Supervisory personnel shall review the data calculations and all QC results.

7.17 The QC data shall be retrievable for all analytical results.

7.18 The method detection limits for all analyses shall be determined and the results documented.

7.19 Computer software programs shall be documented and validated.

7.20 All clients shall be informed if their work is subcontracted and to whom the work is subcontracted.

7.21 All subcontract laboratories shall be evaluated for QA following the guidelines outlined in this practice.

7.22 The laboratory shall perform routine analyses of solvents used for dilutions and extractions to check for contamination.

7.23 The laboratory shall analyze trip blanks as requested by the client or when necessary as indicated by associated samples.

7.24 Chain-of-custody records shall be maintained for all samples and shall be reported with the data when requested.

7.25 The laboratory shall analyze either field duplicates or laboratory duplicates with every group of 20 samples or once per analysis run, whichever is most appropriate (when physically possible given the sample type).

7.26 The precision of the duplicate analyses shall be calculated and the results recorded.

7.27 The laboratory shall have a record of whether it has any history of contamination problems and, if so, what has been done to correct it.

7.28 A reagent or method blank shall be analyzed with every run sequence.

7.29 Spiked samples or blank spikes shall be analyzed once every 20 samples or once per analysis day, whichever is most frequent.

7.30 Blind samples for each analytical procedure shall be analyzed at least quarterly if available.

7.31 Surrogate standards shall be added to all organic samples and organic QC samples whenever possible.

7.32 Blind quality-control samples shall be analyzed at least quarterly by each analyst who performs a given test.

7.33 Training records shall be maintained for all analysts.

7.34 Calibration procedures shall be documented for all test procedures and shall be available to the appropriate personnel.

7.35 Reference standards shall be available and in use as needed. They shall whenever possible be traceable to the National Institute of Standards and Technology (NIST or NBS).

7.36 Quality control check standards shall be analyzed at least once every ten samples with a minimum of one per batch and shall be within the set limits. If the limits are exceeded, all samples analyzed since that last QC standard must be reanalyzed.

7.37 Standard operating procedures (SOPs) shall be available for all test methods and all QC procedures and policies.

7.38 All test samples must be identified with unique identification numbers.

7.39 Only the supervisor (or his designee) in each of the testing areas or their superior shall have the authority to sign test reports or release test data.

8. Facilities and Equipment

8.1 The laboratory shall be controlled with limited access. Limited access being defined as direct entrance by select laboratory personnel with all other access monitored in an appropriate manner.

8.2 The equipment shall be protected from harmful conditions such as exposure to acid fumes, extreme heat, and excessive dust.

8.3 The laboratory environment shall be monitored for proper air flow, ventilation, humidity, and temperature.

8.4 There shall be adequate work space so that each test procedure can be performed safely and efficiently with the least possibility for cross contamination.

8.5 The lighting shall be such that all tests can be performed adequately. For example, titrimetric color changes are easily noted.

8.6 Stable power supplies shall be available. Power regulators shall be used for all major pieces of analytical equipment such as the GC/MS.

8.7 There shall be a source of distilled/demineralized water that has been demonstrated to be free of interferences and contaminants at the necessary detection limits.

8.8 The conductivity of the distilled/demineralized water supply shall be checked daily with the result recorded and shall meet the specifications of the system manufacturer.

8.9 Sufficient exhaust hoods shall be available for volatile/hazardous materials. The hood flow shall be checked at least annually, with a recommendation of every six months.

8.10 Contamination-free work areas shall be present for low-level analyses and microbiological testing.

8.11 Proper work areas, shall be present for handling hazardous chemicals with adequate protection in the case of spillage. This may include the use of stainless-steel trays, plastic trays, or absorbent material.

8.12 Separate cold storage areas shall be available for volatile samples, extracts, standards, reference materials, and other samples as outlined by EPA preservation criteria. The storage areas shall be maintained at $4 \pm 2°C$ and be kept secured/controlled.

8.13 Adequate procedures and facilities shall be available for the collection, storage, and disposal of chemical wastes.

8.14 Proper storage facilities, which are in use, shall be

available for volatile, corrosive, explosive, and flammable materials.

8.15 Testing procedures shall be adequately separated to avoid possible cross contamination due to vapors, aerosols, dust, etc.

9. General Test Equipment Requirements

9.1 Appropriate and up-to-date instrument operating manuals and SOPs shall be made available to the analysts. The manufacturer's recommendations and procedures shall be used unless superseded by the specific test method.

9.2 Analytical balances shall be capable of measuring to meet the requirements of the test procedure.

9.3 The area around the balances shall be appropriately cleaned and free from drafts.

9.4 Evaporation and filtration equipment shall be well cleaned.

9.5 The desiccator and the desiccant shall be in good condition.

9.6 The drying ovens shall be electrically safe and capable of reaching and maintaining the required temperatures. If the temperature of the oven cannot be read without opening the oven door, the thermometer bulb shall be immersed in sand.

9.7 Muffle furnace temperatures shall be achievable as required.

9.8 The pH meter shall have the appropriate electrode with scale graduations of at least 0.1 pH units. A temperature sensor for automatic calibration shall be used or a thermometer for manual corrections shall be in place. The probe shall be stored in accordance with the manufacturer's recommendations when not in use.

9.8.1 The buffer solutions shall not exceed the manufacturer's labeled expiration date and be stored in a polyethylene bottle. The aliquot of used buffer solutions shall be discarded after each days use.

9.9 A magnetic stirrer with a PFTE-stir bar shall be available.

9.10 A conductivity meter and a probe of sufficient sensitivity shall be in use.

9.11 Appropriate glassware shall be available and only Class A glassware used for volumetric measurements. All Class A glassware shall be segregated from all other glassware.

9.12 All refrigerators shall be capable of maintaining the required temperature and shall be monitored.

9.12.1 The thermometer bulb shall be immersed in liquid and the thermometers shall have increments no larger than 1°C.

9.13 *Atomic Absorption Spectrophotometer (AA)*:

9.13.1 The AA shall have a grating, a photomultiplier detector, and adjustable slits. The AA shall also be capable of being adjusting between 190 and 800 nm.

9.13.2 The fuels and oxidants are to be of commercial grade or better.

9.13.3 A filter moisture trap shall be in use between the air source and the spectrophotometer.

9.13.4 The nitrous oxide shall be of reagent grade or better.

9.13.5 Backflash arresters and heaters shall be in place where needed.

9.13.6 Burner head gases shall be removed by ventilation.

9.13.7 All gages and couplings shall be correctly mated.

9.13.8 Proper burner heads shall be available and in use. They will be clean and free of buildup.

9.13.9 For graphite furnace AAs, the tube shall be changed at least every six months or as needed and the chamber cleaned. Performance of this procedure shall be documented in the maintenance log.

9.13.10 Single-element lamps are preferred, but not required. The date when each is first put into use shall be noted.

9.13.11 Background correction capabilities shall be available.

9.13.12 If a cold vapor mercury analyzer or attachment is used, an absorption cell with quartz windows on each end shall be available. In addition, any other equipment required by the method shall be available.

9.14 *Inductively Coupled Plasma (ICP)*:

9.14.1 Background correction shall be available and in use for the ICP.

9.14.2 The nebulizer shall be free of salt buildup and the method used to control this shall be noted. Rinsing with a method blank between samples is the preferred method.

9.14.3 The ICP shall be equipped with an argon gas supply.

9.14.4 Whether the ICP is a sequential or simultaneous element analyzer shall be noted.

9.15 *Visible Spectrophotometer*:

9.15.1 The cell compartment of the spectrophotometer shall be able to accommodate the cell sizes which are needed to perform the specific task.

9.15.2 The cells shall be clean and free of scratches, finger prints, and evaporated film residue.

9.15.3 The lab shall possess at least one pair of matched cells with documented equivalency checks.

9.15.4 The spectrophotometer shall be capable of reading to wavelengths needed to perform the test of interest.

9.15.5 For an automated spectrophotometer, there shall be a chemical drain in place and the following items in use: a sampler, continuous filter, proportioning pumps, analytical cartridges as required, manifolds as required, colorimeter with various filters and flow cells, recorder, heating baths as required, a block digester, and a digital printer. All tubing diameters shall be appropriate for the analyses.

9.16 *Automatic Titrators*—Automatic titrators shall be used in accordance with the manufacturer's instructions and shall be properly maintained.

9.17 *Electronic Probes*:

9.17.1 The meter used shall either have an expanded millivolt scale or read directly in concentration units.

9.17.2 Each electrode used shall be appropriate to the test procedure.

9.17.3 A sleeve-type (non-fiber) or combination electrode shall be available.

9.17.4 The analyst must understand potential interferences for the probes in use.

9.17.5 The meter shall be allowed to warm up for the time recommended by the manufacturer.

9.17.6 For the dissolved oxygen electrode, the membrane shall be changed frequently and the appropriate membranes and electrolyte available.

9.18 *Gas Chromatograph (GC)*:

9.18.1 The GC oven shall be capable of temperature control within ±1.0°C up to a temperature of 300°C.

9.18.2 The following detectors shall be available when necessary and shall be used properly:

9.18.2.1 Electrolytic conductivity,

9.18.2.2 Microcoulometric,

9.18.2.3 Photoionization,

9.18.2.4 Flame ionization,

9.18.2.5 Electron capture,

9.18.2.6 Nitrogen/phosphorus,

9.18.2.7 Flame photometric,

9.18.2.8 Hall, and

9.18.2.9 Thermal-energy analyzer.

9.18.3 If a chart recorder is in use, it shall have a chart width of at least 10 in. (254 mm), be able to give a full-scale response in no more than 1 s, have a signal that matches the instrument, and shall have an adjustable chart speed.

9.18.4 The purge-and-trap system shall be capable of providing finely divided gas bubbles throughout the sample by means of the purge inlet gas device. It shall also be capable of retaining compounds at room temperature.

9.18.5 The desorber for the purge-and-trap unit shall be capable of heating the trapping device to at least 180°C with less than 40°C overshoot.

9.18.6 The purge-and-trap unit shall be capable of accepting 5-mL samples with a gaseous headspace of less than 15 mL.

9.18.7 The trap shall have a length of at least 20 cm.

9.18.8 Appropriate columns for primary and confirmation runs shall be available for the various test parameters.

9.19 *Gas Chromatography/Mass Spectroscopy (GC/MS)*:

9.19.1 It is preferable that all GC/MSs be programmable.

9.19.2 The GC/MS interface shall be glass or glass lined and a split/splitless capillary injection system shall be in place.

9.19.3 The MS shall be capable of scanning from 70 to 450 mass units every 7 s or faster.

9.19.4 The computer system shall be capable of collecting data continuously throughout the entire chromatographic run.

9.19.5 The computer system software should contain the most recent spectral library available.

9.19.6 The computer software shall allow integrating the abundance in any extracted ion current profile between specified time or scan number limits.

9.19.7 In-house replacement parts shall be available for those items that are consumable and often replaced.

9.19.8 Appropriate GC columns shall be available for the specific analysis methods.

9.20 *High-Performance Liquid Chromatography (HPLC)*:

9.20.1 The proper columns and syringes shall be in place.

9.20.2 The HPLC shall contain the detector most appropriate for the compounds of interest.

9.20.3 Some type of recorder or integrator plus a data reduction system shall be in place.

9.20.4 The HPLC system shall have the appropriate injection system.

9.20.5 The laboratory shall possess an electrode polishing kit.

9.20.6 Consistent volume injection loops shall be in use or a system to record the injected volume to the nearest 0.5 µL.

9.20.7 The mobile phase shall be prepared at least weekly and degassed daily.

9.21 *Ion Chromatography (IC)*:

9.21.1 The IC shall contain an anion-guard column, an anion-separator column, an anion-suppressor column, a conductivity cell detector, and either a strip-chart recorder or an integrator.

9.21.2 Particle sizes larger than 0.20 µm shall be filtered from both samples and solutions.

9.21.3 The reagent water shall be free of the anions of interest.

9.21.4 Nitrite and phosphate working standards shall be prepared daily and all other working standards prepared on at least a weekly basis.

9.21.5 The same size sample loop shall be used for both the samples and the standards.

9.22 *Heating Blocks*—Heating blocks shall be capable of achieving and maintaining a temperature of at least 150 ± 2°C.

9.23 *Total Organic Carbon (TOC) Analyzer*:

9.23.1 *Combustion–Infrared*—A means shall exist for the reduction of particle size since sample introduction requires a small particle size. There shall be a separate chamber for the measurement of inorganic carbon. All contact with organic matter shall be avoided before and during the analysis. The carrier gas shall be CO_2 free and contain less then 1 ppm hydrocarbon. The instrument shall be stabilized at 900°C before use and a homogenized blank run before any samples. The syringe size shall be compatible with the particle size in the sample. (IC correction)

9.23.2 *Persulfate–Ultraviolet Oxidation*—The TOC analyzer shall have a nondispersive infrared analyzer along with a flame ionization detector and a chemical titrator. Particle size reduction shall be performed when necessary and glass fiber filters shall be acid washed before use.

9.23.3 *Wet Oxidation*—This method is only applicable for low-level nonpurgeable organic carbon. The potassium persulfate shall be granular and the glass fiber filters acid washed.

9.24 *Total Organic Halide (TOX) Analyzer*—All glassware shall be cleaned with an adequate cleaning solution and muffle furnace fired at 400°C (except volumetric glassware) for 15 to 30 min. The purity of the activated carbon shall be verified before use and the adsorption efficiency checked. The pyrolysis of the sample shall be done in an oxygen-rich environment and the possibility of breakthrough on heavily contaminated samples checked.

9.25 *Pensky Martin Closed Cup Flash Tester*:

9.25.1 The instrument shall not be modified in any way.

9.25.2 Two thermometers shall be present.

9.25.3 Heating of the samples shall be done with care and at a rate that will avoid the loss of sample.

9.25.4 The flash point shall be adjusted for barometric pressure.

9.26 *Setaflash Closed Cup Flash Tester*:

9.26.1 Heat transfer tape shall be in use and the test apparatus placed in subdued light and out of disturbing drafts.

9.26.2 A magnifying glass shall be available to read the thermometer.

9.27 *Extraction Procedure (EP) Toxicity Test Apparatus*:

9.27.1 The extractor shall be built in accordance with the suggested design and allow for mixing such that stratification of the sample does not occur.

9.27.2 The extractor shall be maintained such that the sample is rotated at a rate of 20 rpm.

9.28 *Toxic Characteristic Leaching Procedure (TCLP) Apparatus*:

9.28.1 *Agitation Apparatus*—The extraction vessel shall rotate end-over-end at a rate of 30 ± 2 rpm with the temperature of the area monitored and recorded.

9.28.2 *Extraction Vessel (TCLP)*—The extraction vessel shall be made of plastic-coated borosilicate glass and have a volume of 2 L.

9.28.3 *Extraction Vessel (Zero Head Extractions (ZHE))*—The ZHE shall be a commercial device that is pressure checked for leaks after each use. The vessels shall be made of either glass, PTFE, or 316 stainless steel. HDPE, PVC, or polypropylene devices are to be used only for metals mobility tests.

9.29 *Autoclaves*:

9.29.1 The autoclave shall be capable of reaching a sterilization temperature of 121°C, maintain that temperature for no more than 15 min, and require no more than 45 min for a complete cycle.

9.29.2 Temperature and pressure gages shall be on the exhaust side on a flow-through autoclave along with an operating safety valve.

9.29.3 The autoclave shall depressurize at a slow enough rate so that the culture media do not boil over.

9.30 *Ultraviolet (U.V.) Sterilizer*—The sterilizer shall be properly disinfected before each use and the bulb replacements done on a routine basis.

9.31 *Incubators*:

9.31.1 The incubators shall be large enough to prevent overcrowding of the samples and have an internal temperature-monitoring device sensitive to ±0.5°C.

9.31.2 The temperature shall be maintained at the appropriate temperature for the microbiological test being performed.

9.32 *Water Baths*:

9.32.1 The water baths shall be large enough to prevent overcrowding of samples.

9.32.2 Circulating water baths are recommended for all intended uses but are only required when microbiology samples are being prepared. Any other water bath such as for mercury digestions may be noncirculating.

9.33 *Filtration Equipment*:

9.33.1 The filtration unit shall be made of a material suitable for the test procedure and be used with the appropriate size and type of filter.

9.33.2 If pressure filtration is used, the pressure shall never exceed 50 psi.

9.34 *Radiochemistry Equipment*:

9.34.1 All detectors shall be stored in graded lead shielding.

9.34.2 The instruments shall be in a room separate from where samples and standards are handled and prepared for analysis.

10. Reagents

10.1 All chemicals and reagents shall be labeled, dated, and signed with the date of receipt or the day the reagent is prepared.

10.2 All chemicals and reagents shall be proven free of contaminants and interferences.

10.3 All acids shall be of reagent grade or better, except for those used for ICP work, which need to be of high-purity grade or equivalent.

10.4 All solvents shall be of chromatographic grade or better.

10.5 A log book shall be maintained of all reagents.

11. Calibration

11.1 A program shall exist for initial and periodic calibration of all equipment such that the frequency, conditions, standards, and calibration history are documented.

11.2 All reference material and where applicable, all measurements shall be traceable to an appropriate agency, this includes calibration standards. Document the preparation. The documentation shall include the solvent, concentration, date, preparer's name, and the expiration date.

11.3 Use only primary reference standards for calibration.

11.4 Verify all working standard concentrations versus the primary standard and document the comparison.

11.5 Calibration protocols for all the analytical instrumentation shall be available to the analysts.

11.6 Keep all calibration results in a permanent record.

11.7 Whenever possible, use a quality-control sample to verify calibration standards.

11.8 Keep maintenance logs on each piece of analytical equipment and recalibrate all equipment following any type of repair or when the performance of the equipment is in doubt.

11.9 *Analytical Balances*:

11.9.1 Check each analytical balance daily (or with use) with a minimum of one Class S or S-1 weight in the range in which the measurements will be made. Check the balance monthly with a series of Class S or S-1 weights and document the results. Any variance of greater than 0.1 % between the expected weight and the actual weight requires corrective action.

11.9.2 At least annually, calibrate the balance by a certified technician and document the calibration.

11.10 *Class S Weights*—Calibrate the Class S weights that have been calibrated within the last five years and are traceable to NIST (NBS).

11.11 *Drying Ovens*—Check the temperature of each oven before and after each usage to verify the correct operating temperature for the given test procedure. Document all checks in a bound logbook.

11.12 *Muffle Furnace*—Verify the temperature of the muffle furnace at least annually in the range in which it is operated and document the verification.

11.13 *pH Meter*:

11.13.1 Perform a visual check of the probes and document before each use. Note hairline cracks, plugged fiber tips, loose sleeves, and low reference solution and replace.

11.13.2 Use either NIST (NBS) primary buffer salts to prepare buffer solutions or commercial secondary buffer solutions traceable to NIST (NBS).

11.13.3 Standardize the pH meter at least daily with

twobuffers, 7.0 standard pH units and normally 4.0 standard pH units. However, make the standardization to bracket the expected pH range. This bracket must be no more than 3 to 4 pH units. Document the standardizations in a bound notebook.

11.13.4 Restandardize the pH meter following the analysis of any very acidic (<2 pH units) or very basic (>12 pH units) sample.

11.14 *Conductivity Meter*:

11.14.1 A probe of sufficient sensitivity.

11.14.2 Perform a daily or before-use calibration check and document the results.

11.14.3 Determine the cell constant of the probe on an annual basis.

11.15 *Refrigerators*—Check the temperature of each refrigerator in use at least twice every working day and record the temperature. The temperature of a sample storage unit shall be between 2 to 6°C with the target temperature of 4°C.

11.16 *Thermometers*:

11.16.1 Check the calibration of each mercury or alcohol thermometer in use at least annually against an NIST (NBS) traceable thermometer. This check shall be at two separate temperatures and document the results.

11.16.2 Calibrate dial-type thermometers at least quarterly against an NIST (NBS) traceable thermometer.

11.16.3 Label the thermometers in some fashion so as to note the latest calibration date and any correction required.

11.17 *Atomic Absorption Spectrophotometer*:

11.17.1 Analyze a standard calibration curve made up of a method blank and at least three levels of standards covering the concentration range of the samples daily with each sample batch.

11.17.2 Verify the standard curve with the blank and at least one standard once every 20 samples. The verification shall be within 10 % of the original calibration.

11.17.3 Prepare a calibration curve for each of the elements being analyzed.

11.18 *Inductively Coupled Plasma Spectrometer*:

11.18.1 Analyze a calibration blank at the beginning of each run sequence and once every 20 samples thereafter.

11.18.2 Analyze a reagent blank with each group of 20 samples.

11.18.3 Analyze a standard curve daily consisting of a method blank and five levels of standards covering the concentration range of the samples. The actual standard values shall not deviate by more than 5 %.

11.18.4 Before any set of samples is analyzed, reanalyze the highest concentration calibration standard.

11.18.5 Flush the ICP system with the calibration blank between each standard analysis.

11.18.6 Determine instrument drift with each analysis run and document the results.

11.18.7 Check background correction factors before, during, and after each analysis run.

11.19 *Visible Spectrometer*:

11.19.1 Check wavelength accuracy and repeatability of the instrument at least quarterly at several wavelengths using a holmium oxide glass filter. Document the results.

11.19.2 Check the photometric accuracy and repeatability of the instrument at least quarterly using a blue glass filter traceable to NIST (NBS) and document the results.

11.19.3 Check matched cells for equivalency at least every six months.

11.19.4 Analyze a calibration curve consisting of a method blank and at least three standards that bracket the range of the sample concentrations with each batch of samples and verify the calibration every 20 samples with at least one standard.

11.20 *Automated Spectrophotometer*:

11.20.1 Obtain a stable baseline with all the reagents while distilled water is fed through the sample line.

11.20.2 Arrange standards for a phosphorus analysis in decreasing order of concentration.

11.20.3 Analyze standards for a TKN analysis in the order of increasing concentrations.

11.20.4 Run a minimum of three standards and one method blank with each autosampler tray of samples.

11.20.5 For nonlinear type analyses, analyze a minimum of four standards and a method blank.

11.21 *Autotitrators*—Check the accuracy of the autotitrators versus a Class A pipet by means of weight determinations at least quarterly and the results documented. If more than a 5 % difference between the two methods is found, appropriate corrective action must be taken.

11.22 *Electronic Probes*:

11.22.1 Use buffers of high ionic strength where appropriate.

11.22.2 Prepare a standard curve consisting of a method blank and at least five standards at concentrations that bracket the sample concentrations at least every six months. This curve shall also be prepared any time new reagents are used for the sample's preparation.

11.22.3 Prepare a standard curve versus millivolt readings for each electrode in use.

11.22.4 Reconfirm the calibration at one concentration after each sample measurement. Document this confirmation in the laboratory notebook.

11.22.5 Immerse the electrode in the sample and the standard for an equal period of time before a reading is taken.

11.23 *Gas Chromatograph (GC)*:

11.23.1 Analyze a minimum of one method blank and one mid-point calibration standard daily and verify with at least one standard every 20 samples and at the end of the analysis run. The calibration standard shall contain all the analytes of interest.

11.23.2 At least quarterly, the laboratory shall analyze a QC check standard for each contaminant of interest. Document these results.

11.23.3 Store all standards with minimal headspace and check frequently for degradation.

11.23.4 Purge the system daily before any sample analyses.

11.23.5 If the calibration acceptance criteria is not met, reanalyze the calibration curve a second time. If the curve still does not meet acceptance criteria, alternate corrective action must be taken.

11.23.6 If acceptance criteria have not been met, the QA check sample must be reanalyzed when the problem is corrected to verify that the requirements are met.

11.24 *Gas Chromatograph/Mass Spectrometer (GC/MS)*:

11.24.1 The mass spectrum of decafluorotriphenyl phosphine or bromofluorobenzene must meet the instrument manufacturer's specifications and the appropriate regulatory guidelines for these specific tuning compounds. Document the results of these analyses.

11.24.2 Replace the stock standard solutions at least every six months and sooner if degradation or volatilization is noted.

11.24.3 Analyze a standard curve consisting of a method blank and at least five calibration standards at least quarterly. Each standard shall contain all the analytes of interest. Perform the calibration more frequently depending on the sensitivity and stability of the instrument.

11.24.4 Verify the calibration at the beginning and end of each analysis, once every 20 samples, and once during every 12 h of continuous operation. From this check the relative standard deviations for specified analytes and the retention times of all analytes reviewed.

11.24.5 From the initial calibration information, all criteria must be met as outlined by the instrument manufacturer and the appropriate regulatory agency. This includes the relative response factor for each of the analytes and the relative standard deviation of the responses over the full range of the curve.

11.24.6 The internal standard calibration procedure is recommended for most analyses. Make a note if an alternative method is used.

11.25 *High-Performance Liquid Chromatograph (HPLC)*:

11.25.1 If either the external calibration system or the internal calibration system is used, calibrate the HPLC system with a minimum of three concentration levels that bracket the sample concentrations for each analyte of interest.

11.25.2 If the internal calibration system is used, it shall be demonstrated that the measurement of the internal standard is not affected by the method or matrix interferences.

11.25.3 Document all response factor information.

11.25.4 Verify the calibration curve or the calibration factor each working day by analyzing one or more calibration standards. Document this verification.

11.26 *Ion Chromatograph*:

11.26.1 Prepare a calibration curve for each of the analytes of interest with a minimum of three to five concentration levels spanning the expected concentration range of the samples and a method blank. Reanalyze the calibration curve at least every six months and more often if a problem arises or new reagents are used.

11.26.2 One of the points on the curve shall be near the method detection limit as appropriate.

11.26.3 Document the retention times of each of the analytes from the calibration run.

11.26.4 Verify the calibration curve each working day by the analysis of one of the calibration standards. If more than 20 samples are analyzed, reverify the calibration curve every 20 samples. If the results of the verification standard vary by more then ±2 to 3 times the RSD from the original curve, redo the verification with a fresh calibration standard.

11.27 *Heating Blocks*—Test the temperature range of the heating block when first put into service. Monitor the temperature each time the heating block is used and the record results in a laboratory notebook.

11.28 *Total Organic Carbon (TOC) Analyzer*:

11.28.1 Calibrate the TOC analyzer before each use and verify the calibration at least once every 20 samples.

11.28.2 For the persulfate-ultraviolet oxidation method, check the oxidation efficiency before each use and document the check.

11.29 *Total Organic Halide Analyzer*:

11.29.1 Prepare a calibration curve consisting of a minimum of three standards that bracket the expected sample concentrations at least every six months. Rerun the curve more often as needed and any time new reagents are introduced.

11.29.2 Run duplicate instrument calibration standards and duplicate blank standards before the first sample analysis on each day of use.

11.29.3 Reanalyze the calibration standard and blank once every 20 samples.

11.30 *Flash Point Tester*:

11.30.1 These calibration guidelines are for both the Pensky-Martens Closed Cup Flash Tester and the Setaflash Closed Cup Flash Tester.

11.30.2 Use p-Xylene as the reference standard to check the equipment.

11.30.3 Check the reference standard in duplicate before analysis of any samples and once every 20 samples thereafter.

11.31 *Extraction Procedure (EP) Toxicity Apparatus*:

11.31.1 Calibrate the rotation speed of the extraction apparatus and document before the extractor is first put into use and then recheck annually.

11.32 *Toxic Characteristic Leaching Procedure (TCLP) Apparatus*:

11.32.1 Set the rotation speed of the extractor at 30± 2 rpm and check and document the speed.

11.32.2 Check the interior of the zero head extractor for scratches or gouges. If any are found, do not use the apparatus.

11.33 *Autoclaves*—Before the initial use and once every quarter verify the temperature and the pressure in the autoclave and document the results.

11.34 *Incubator*—Set the temperature of the incubator at the temperature appropriate for the microbiological analysis being performed and check the temperature twice per working day and document the checks.

11.35 *Water Baths*—Maintain the temperature of the water baths at the correct temperature for the method in use. Stabilize the temperature before use and then monitor with a thermometer throughout the time its in use. Document these checks in a laboratory notebook.

11.36 *Liquid Scintillation Counters*:

11.36.1 Before any calibrations are performed, check the zero-pole and baseline offset of the amplifier output.

11.36.2 Prepare efficiency and background control charts at least every six months.

11.36.3 Prepare a calibration curve consisting of three standards and one method blank at least every six months.

11.36.4 Verify the calibration and window settings with one standard at the beginning of each day of use and once every 20 samples. Document this check.

11.36.5 Analyze a quench sample at the beginning of each sample batch.

11.37 *Gas Flow Proportional Counters*:

11.37.1 The gas flow proportional counters include testing for gross alpha, gross beta, radium-226, radium-228, strontium-89, strontium-90, cesium-134, and iodine-131.

11.37.2 Check the operating voltage plateaus and crosstalk between the alpha and beta channels every time there is a change in the counting gas or any other changes are made to the instrument.

11.37.3 Prepare a calibration curve consisting of a minimum of three standards and one method blank at least once every six months and more often as necessary.

11.37.4 Verify the calibration at the beginning of each batch and once every 20 samples. The check is to document that the gas flow proportional counter is providing Poisson distributed counts.

11.37.5 Check the alpha and beta background count at the beginning of each batch and once every 20 samples. This is a check on the sensitivity of the radioanalysis.

11.38 *All Other Radiochemistry Equipment*:

11.38.1 Calibrate the equipment with a minimum of three counting standards and one background sample at least every quarter. Perform calibrations more often as necessary.

11.38.2 Verify the calibration at the beginning of each batch of samples and once every 20 samples thereafter.

11.38.3 Check the efficiency calibration and the energy calibration over a range of energies once every quarter.

11.38.4 Check the spectrometer's high-voltage daily and compare to the previous days values.

12. Test Methods and Procedures

12.1 Store all samples in a refrigerated area at 4°C until analysis. Store volatile samples in a separate refrigerator to help avoid contamination of the volatile samples. Metal samples may be stored at ambient temperatures.

12.2 Use the appropriate test methodology relative to the compounds of interest, the sample media, and to satisfy the necessary regulatory agencies.

12.3 All analytical data shall go through a minimum two-stage review with one person being the analyst and the second being the area supervisor. Additional reviews are advisable but not required. Complete all reviews at this level before the test report is written. A review of the final report versus the laboratory worksheets should also be performed. This is to help eliminate transcription errors.

12.4 Record all analytical data with any backup information such as chromatograms, calculations, or spectra.

12.5 Each analyst performing a specific test method must have shown proficiency with the test method through the analysis of a QC check sample. Such proficiency must be documented.

12.6 Any deviations from a given test methodology must be documented along with any information that indicates the deviation does not alter the test results.

12.7 Method validation studies must be performed and the results documented whenever a revision to a test procedure is made or a new test procedure is used.

12.8 A procedure shall be in place for the selection, identification, handling, preparing, and storing of all samples.

12.9 Samples should be retained for a minimum of 30 to 60 days after a test report is sent. The storage shall be such that the integrity of the sample is preserved as much as possible.

12.10 Dispose of all samples in accordance with federal and state regulations.

12.11 A procedure shall be in place for making and controlling revisions to in-house standard operating procedures.

13. Records

13.1 There shall be a system in place that provides for retrievability and traceability of the sample source (client), the methodology of each analysis, results (including calibration and instrument checks), the name of the person performing the analysis, the date, and any oddities that may have been noted during analysis.

13.2 Store the records and reports in a secure area for the required amount of time. This time frame is dependent on the various regulatory agencies, but shall be a minimum of three years from the date the report is originally issued.

13.3 All current reference documents shall be available to the analysts. This may include EPA manuals (see SW 846 and Standard Methods), the Code of Federal Regulations (CFRs), ASTM, etc.

13.4 File all laboratory notebooks, when full, along with the associated raw data, QC data, analytical reports and any other pertinent information in a secure area that has a means for retrieval.

13.5 Each laboratory notebook shall have a unique number clearly displayed on the cover or the spine. These numbers are used for control of the notebooks assigned. Keep a record that contains the name of the person receiving the notebook, the data issued, date returned, and the place of storage after the book is returned.

13.6 Permanently bind each notebook with consecutively numbered pages or some equivalent.

13.7 All notebook entries must be legible and in ink. Any corrections must be made without obliterating the original information. The original entry shall have a single line drawn through it and the date and initials of the person making the correction noted. There shall be no use of white-out (correction fluid).

13.8 The supervisor of each person maintaining a notebook shall periodically review each notebook and sign and date the review.

13.9 All notebook entries shall be signed and dated by the person making the entry. The signature shall be legible.

13.10 Any space remaining at the bottom of a page that is not intended for immediate use shall have a line drawn through it and the person responsible, initial along the line.

14. Test Reports

14.1 Each test report shall accurately, clearly, and unambiguously present the results and all other relevant information for the sampling event.

14.2 Report, as applicable, the following information:

14.2.1 Name and address of the laboratory,

14.2.2 Unique report identification (including each page),

14.2.3 Name and address of the client,

14.2.4 Sample identification and description,
14.2.5 Date of sample receipt,
14.2.6 Date test performed,
14.2.7 Statement that the test results relate to the tested items only,
14.2.8 Identity of the test method used,
14.2.9 Description of sampling procedure, where relevant,
14.2.10 Any modifications to the test method,
14.2.11 Disclosure of any subcontractor used,
14.2.12 Results,
14.2.13 Any problems encountered during the test,
14.2.14 Any deficiencies in the quality-control system,
14.2.15 Measurement uncertainty (if relevant),
14.2.16 Identity of person accepting responsibility, and
14.2.17 Statement controlling report production except in entirety.

14.3 A method shall exist for suitably marking a test report that is reissued due to corrections or additions.

14.4 Issue each test report only to the client or another individual designated by the client.

14.5 All analytical data, reports, and client files must be maintained in a secure fire-resistant area with limited access.

15. Keywords

15.1 calibration; chemical testing; contained fluids; environmental; equipment; facilities; laboratory management; minimum requirements; organization; personnel; quality systems; rock; soil; test reports

SUMMARY OF CHANGES

This section identifies the location of changes to this specification since the last edition.

(1) Section 5 was deleted and subsequent sections were renumbered.